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Viton® GLT O-Ring Resilience Study

15 March 2001

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Dennis E. Lileikis

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14. ABSTRACT

As part of an ongoing study of the resilience properties of O-ring materials made of Viton®, the properties of Viton GLT were compared with those of Viton B under comparable conditions. This study was the result of a contractor-proposed change in O-ring seal material. United Technologies Chemical Systems Division had proposed a change in the O-ring seal material used on the Titan solid rocket motor (SRM) clevis joint from Viton B to Viton GLT, a fluorocarbon elastomer with better low-temperature properties, in order to eliminate application of external joint heaters.

The O-ring material made with Viton GLT was found to have significantly improved resilience and compression set properties under all conditions studied when compared to Viton B. Even under worst-case conditions, these properties exceeded even the best values obtained in a comparison study with Viton B. Based on these observations, Viton GLT is an excellent candidate for use in demanding applications that require both optimum resilience and compression set properties.

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fluorocarbon elastomer, compression set, aging, solid rocket motor, joint seal

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1. Introduction

The Presidential Commission on the Shuttle Challenger accident on mission 51-L in January 1986 concluded that the cause was the failure of the pressure seal in the aft field joint of the solid rocket motor. The Commission also stated that the elastomeric seals were severely affected (hardened) by the low temperatures at launch.[1] Although processed differently, the Shuttle and Titan O-rings were both made of Viton®, a fluorocarbon elastomer. Among the original design requirements for the Titan Solid Rocket Motor (SRM) O-ring seal was a minimum 17% compression squeeze, operating temperatures from 40–90°F, and a 12-month assembled life. As a result of the Challenger accident, joint heaters were evaluated by Titan and applied to SRM joints so that O-ring temperatures were maintained above 60°F. A cross-section of the SRM field joint design, including the joint heater and additional insulation features, is shown in Figure 1.

The Titan IV SRM consisted of seven D6AC steel-alloy case segments and two closures that were mechanically fastened using a clevis-tang joint. Each of the resulting eight field joints used a single continuously molded O-ring. The clevis-tang O-ring joint was designed to accommodate the structural deflections arising during ignition, liftoff, and flight. The O-rings had to be resilient enough to respond rapidly to the maximum expected gap opening due to joint motions resulting from these vehicle deflections.[2] Additional features shown in Figure 1 are as follows:

- insertion of shims between the outer clevis leg and the tang to limit the amount of joint rotation and reduce gap opening potential
- application of DC 55 grease to the joint to provide lubrication to the O-ring seal and to protect bare metal surfaces from corrosion
- application of putty to each face of the insulation as a sealant where joint assembly produced a compression fit between insulation at the tang and at the inner clevis leg (this compression fit results in the putty being exuded where the segments meet)

The igniter flamefront ignites the segment's propellant surfaces, resulting in a rapid pressure build-up to the maximum expected operating pressure (MEOP). During this time, the steel membranes of the case barrel outward to produce roll motion to the joint, which, in turn, pushes the tang and inner clevis leg apart. The pressurized gas penetrates the putty and through multiple charging paths pressurizes the joint area, pushing the O-ring into place. An illustration of the gap that is generated by this joint motion is shown in a schematic of the clevis joint in Figure 2.

Structural analysis of clevis joint motion had predicted that a gap can be generated at the clevis joint where the O-ring is seated. Any gap that develops must be closed by O-ring expansion to maintain the seal during booster lifetime. This expansion, or the recovery property of the O-ring, must take place rapidly enough to seal the clevis joint in the initial 0.5 s, the point at which the vehicle's MEOP is reached.

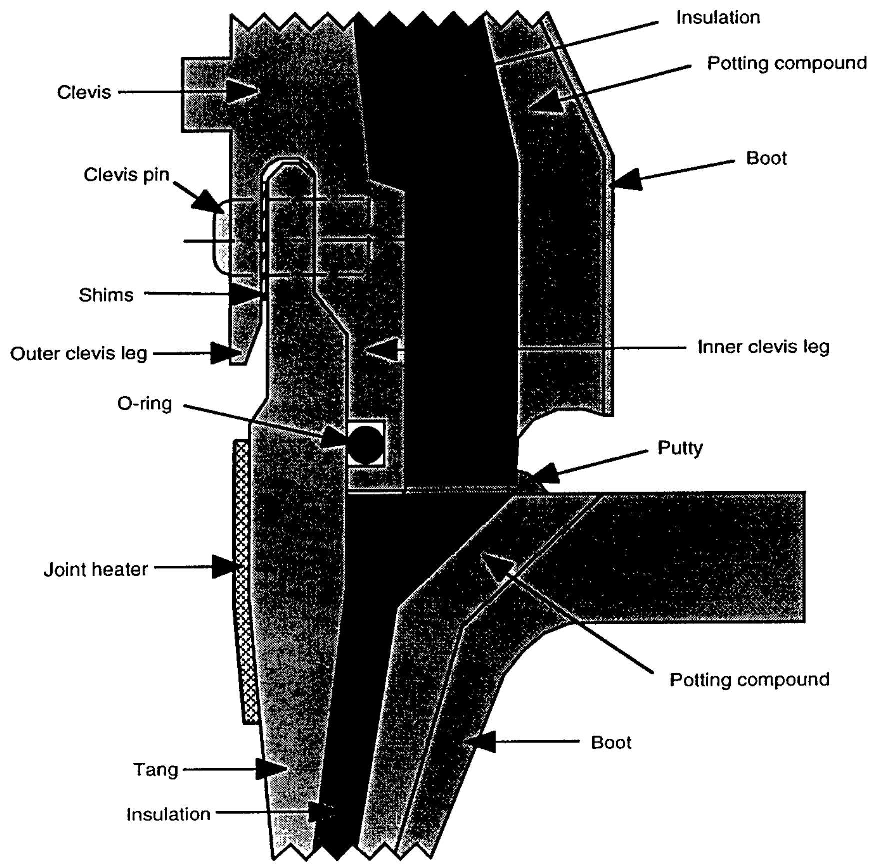


Figure 1. Cross-section of Titan IV SRM field joint assembly with joint heater addition.

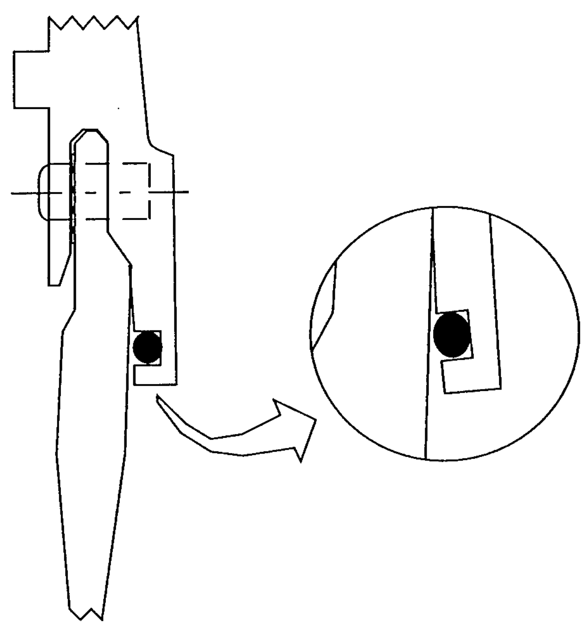


Figure 2. Cross-section of field joint and enlarged area of O-ring region exhibiting deformation at the joint.

Performance of the O-ring is critical to joint seal integrity. Seal performance depends both on the deflection and the deflection rate. Resilience of an elastomer is defined as "the ratio of energy output to energy input in a rapid or instantaneous full recovery of a test piece." [3] Resilience of the O-ring material defines its ability to respond to any gap generated during launch.[4] (Resilience here describes the capability of the O-ring material to respond to joint motion.)

Slow compression-recovery (i.e., slow "rebound" on abrupt release from compression) can result in leakage past an O-ring should separation (gap opening) of the two components being sealed occur at a rate exceeding the recovery rate of the O-ring material.[5] Among the many materials originally evaluated for the demanding seal application at the field joint, Viton B met or exceeded most of the specified properties including long shelf life.[6] In a study of a number of elastomeric materials, it was observed that no material changes in tensile strength, elongation, and compression deflection characteristics occurred in Viton B formulations after 7 and 12 years of shelf life aging.[7]

In a previous report,[8] the resilience of O-ring materials made from Viton B was measured over various time periods under 17% and 23% compression at 60 and 70°F. That report concluded that the resilience of Viton B material was very sensitive to temperature and time under compression. Resilience was found to be significantly reduced with increasing time under compression, which is of concern for delayed launches when segments are stacked for unpredictably long periods. The greatest rate of change in O-ring resilience occurred within the first three months under compression. After three months, the rate of change in O-ring resilience continued at a much lower rate throughout the 36-month test period. Variability in properties among different O-ring batches made from Viton B was also found to be a factor to consider when evaluating sealing ability under specific operating conditions.

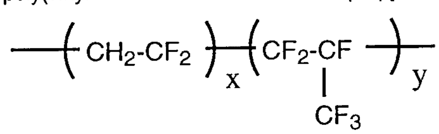
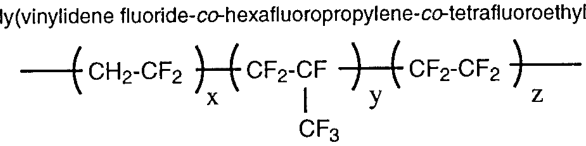
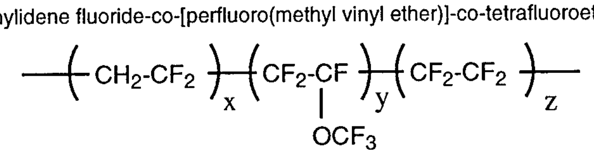
Because the prelaunch environment could expose the O-ring to temperatures below that considered safe for sealing purposes, heaters were attached to the surface of the vehicle at the joints to provide a margin of safety. To eliminate application of the external heaters, Viton GLT, a new candidate material with better low-temperature properties, was proposed as a replacement for Viton B as an O-ring material. Viton GLT is derived from a different family of fluorocarbon elastomers, and each family consists of a number of formulations designed to exhibit specific properties for different applications.

The purpose of this report is to present data for O-rings made of Viton GLT, which was developed to provide improved resilience (recovery) characteristics at lower temperatures when compared with Viton B. Samples from an O-ring made from Viton GLT were evaluated at the same compression values and temperatures as was Viton B for periods of up to 18 months. The O-ring used also had a 115.4-in. inner diameter and a 0.275-in. cross section, similar to the O-rings made from Viton B. Since it was impractical to simulate recovery tests of the actual O-ring because of its size, an alternate experimental procedure was used to test samples on a much smaller scale for extended periods. The experimental procedure used in the previous report for O-rings made from Viton B was also applied to the O-ring made from Viton GLT. Results for Viton GLT are presented and compared, where appropriate, with the results obtained for Viton B in the previous report.

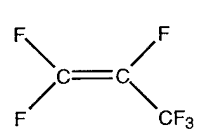
2. Background

In addressing the need to survive hostile environments, primarily fuel and thermal resistance, in addition to low-temperature service, early work on fluorocarbon elastomers led to development of Viton A, a copolymer of vinylidene fluoride (VF_2) and hexafluoropropylene (HFP), which is cured with diamine curing agents. Further improvements in polymer properties resulted in development of the Viton B family, a terpolymer system that contained VF_2, HFP, and tetrafluoroethylene (TFE). Various combinations of VF_2, HFP, and TFE, allow tailoring of the material properties for specific applications. Introduction of bisphenol-based curing agents resulted in an additional compounding variable with major effects on the physical properties of vulcanizates of Viton. Further advances in the specialty category of the Viton families of resins resulted in Viton GLT, which is a terpolymer of VF_2, perfluoro(methyl vinyl ether) (PMVE), and TFE. Viton GLT was found to have improved low-temperature performance with the added capability of being crosslinked with a peroxide cure system.[9] The chemical names and structures for these fluorocarbon elastomers are described in Table 1.[10]

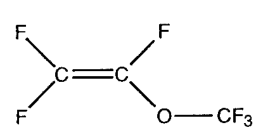
Table 1. Commercial Fluorocarbon Elastomers

Copolymer (Name/Structure)	Commercial Designation
poly(vinylidene fluoride-*co*-hexafluoropropylene 	Viton A
poly(vinylidene fluoride-*co*-hexafluoropropylene-*co*-tetrafluoroethylene) 	Viton B
poly[vinylidene fluoride-co-[perfluoro(methyl vinyl ether)]-co-tetrafluoroethylene] 	Viton GLT

The primary distinction between the backbone structures of Viton B and Viton GLT is the difference in their comonomers, HFP and PMVE, shown below.



Hexafluoropropylene (HFP)



Perfluoro(methyl vinyl ether) (PMVE)

The spatial relationship of the trifluoromethoxy pendant group in PMVE compared to that of the trifluoromethyl pendant group in HFP, when part of a polymer chain, affects the interchain packing capability of the polymer. This usually results in greater chain flexibility and better low-temperature properties. This is evidenced by a glass-transition temperature (T_g) near –40°C for GLT, while that for Viton B is ordinarily found in the range of –20°C.[11] This is to be expected because the HFP monomer polymerizes to a homopolymer having a T_g of 165°C, while the homopolymer of PMVE has a T_g of –25°C. Thus, highly fluorinated copolymers containing PMVE with partially fluorinated monomers can be prepared that have high fluorine content but a lower T_g. The effect of composition on the low-temperature properties of terpolymers of PMVE, VF_2, and TFE, is illustrated in Figure 3.[12] For example, a terpolymer of VF_2, TFE, and PMVE in the approximate molecular ratio of 75/10/15 has a T_g of –37°C.

The presence of oxygen in the GLT terpolymer can also contribute to differences in chemical and physical properties from those in Viton B. Significant differences are found between families of Viton in terms of resistance to volume change or property degradation. These differences are particularly notable in low molecular weight, oxygenated solvents, or in very aggressive lubricants (containing highly basic additives).[9] Examples of these property differences are seen in Table 2.

As can be seen in Table 2, the difference in volume change between Viton B and Viton GLT is negligible when exposed to a hydrocarbon fuel. However, a significant difference in volume change is observed when exposed to the small oxygenated methanol solvent. The presence of the oxygenated substituent in Viton GLT assists the ability of the polymer to absorb polar molecules more readily than does Viton B. This effect is much like plasticization or softening of the material. Although both families of fluorocarbon elastomers can be considered highly resistant to degradation with excellent elastomeric properties, subtle property differences may be important in specific applications as indicated in Table 2.

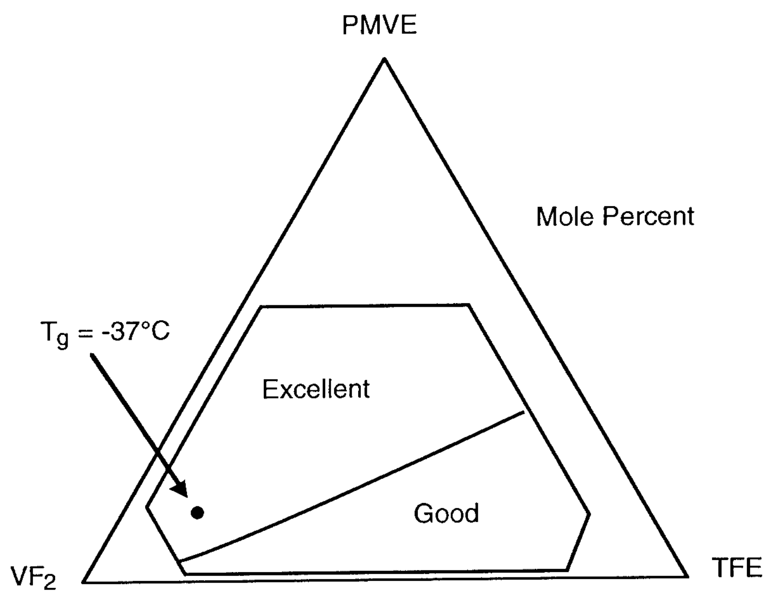


Figure 3. Low-temperature flexibility of cured terpolymers of PMVE, VF_2, and TFE.

Table 2. Typical Physical Properties of Viton B and GLT Fluorocarbon Elastomer Families

Property	B	GLT
Nominal fluorine content, wt. (%)	68	64
Percent volume change in fuel C, 168 h at 23°C	3	5
Percent volume change in methanol, 168 h at 23°C	40	90
Low-temperature flexibility, TR-10, °C (ASTM D 1329)	−13	−30

The diamine, bisphenol, and peroxide crosslinking agents used to cure the fluorocarbon elastomer gum do so by different crosslinking mechanisms. This factor, in addition to differences in reaction rates, leads to different properties. The crosslinking site itself often is a major contributor to these property differences and must be understood to appreciate its importance.[13] A comparison of the effects of different curing agents on some properties of Viton elastomers is shown in Table 3.

As can be seen from Table 3, the choice of curing system can produce very different kinds of properties and usually depends on the end-use application. For example, diamines produce materials with poor compression set resistance but offer unique high hot tensile strength and good adhesion to metals. The bisphenols provide faster cure rates and good resistance to compression set, while peroxide curing systems are also fast curing, but result in intermediate compression set values.[9]

Fluorocarbon elastomer gums can be obtained with an incorporated-cure system where only fillers need be added for a complete formulation, or as the raw gum, allowing the rubber formulators to tailor the curing system to their specific requirements. In O-ring applications, the primary consideration is resistance to compression set. A fluorocarbon elastomer gum is chosen based on molecular weight, crosslink density, and cure system to provide the best combination of processibility and end use performance.[14] A typical example of a fluorocarbon elastomer compounding scheme is shown in Table 4.

In a typical formulation for a material qualifying for Mil-83248-1, that designated for the clevis O-ring, approximately 9 phr of a combination of the inorganic bases and 30 phr of MT Black are used. Careful compounding and processing of the fluorocarbon elastomer gumstock are critical to formation of a material with the desired performance characteristics. Batch-to-batch variations can easily occur and material property characterization must be performed to ensure product conformance.

Table 3. Comparative Performance of Viton Materials from Different Curing Systems

Property	Cure System		
	Diamine	Bisphenol	Peroxide
Fast cure rate	P–F	E	E
Adhesion to metal inserts	E	G	G
Compression set resistance	P	E	G
Steam, water, acid resistance	F	G	E
Flex life	G	G	G

Rating: E = excellent G = good F = fair P = poor

Table 4. Typical Fluorocarbon Elastomer Compound

Component	Amount (phr)*
rubber (may include curative)	100
inorganic base: magnesium oxide, calcium hydroxide	6–20
filler (reinforcing or nonreinforcing)	0–60
accelerators or curatives (if not included in the base rubber)	0–6
process aids	0–2

*parts (wt.) per hundred of rubber

3. Experimental

One-in.-long samples were cut from an O-ring made from Viton GLT. Each sample was subjected to one of two levels of compression (17% and 23% squeeze), the minimum and maximum values allowed within the Titan specification, and comparable with data obtained from a previous NASA study of O-rings. The sample holder consisted of a 2 × 2 × 1-in.-thick aluminum block. A channel was machined that approximated the size of the O-ring groove of the clevis joint. A flat aluminum cover piece was machined to provide the mating surface. For measurement purposes, a metal strip slightly longer than the length of the sample and 0.008-in. thick, to maintain planarity, was bonded to the Viton GLT sample using a thin layer of epoxy adhesive. A small hole was placed in one end of the metallic strip to provide an attachment location for the linear variable differential transformer (LVDT). The total thickness of the sample, including the adhesive and metallic strip, was measured, and the sample was placed in the groove with the metallic strip facing upward. A series of metal shims, each 0.001-in.thick, were used in combination with the measured depth of the sample holder channel to approximate either the 17% or 23% compression squeeze. The shims were placed between the aluminum block, and its cover piece and the segments were bolted together. An illustration of the sample configuration and parts used to construct the sample holder is shown in Figure 4.

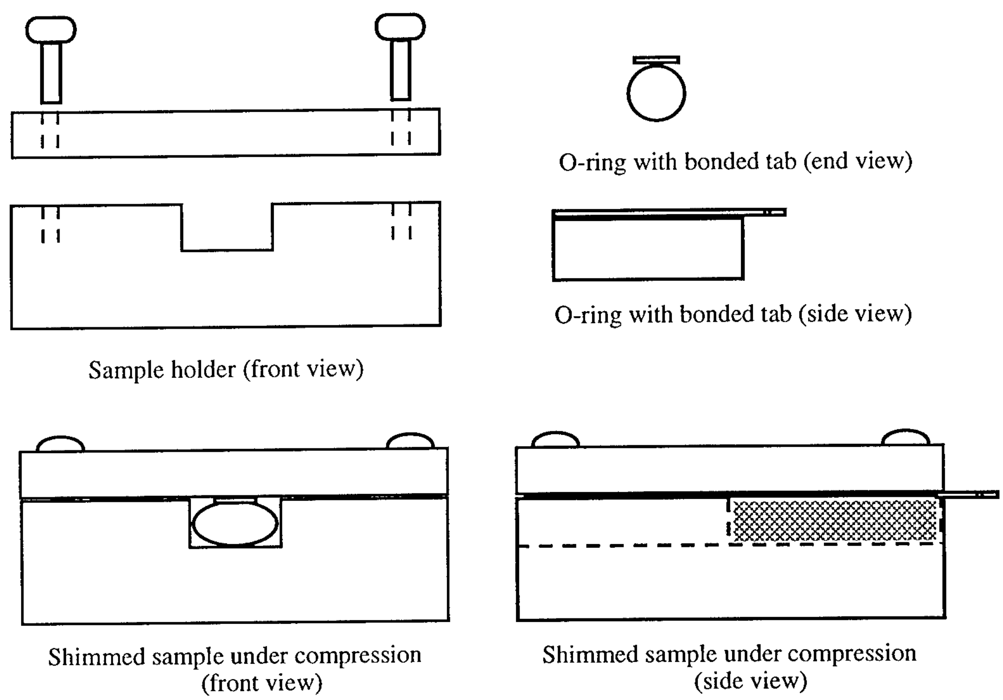


Figure 4. Example of O-ring configuration and sample holder construction.

The samples, configured as shown in Figure 4, were stored under ambient conditions for specific time periods. At the end of a selected time period, the samples were tested at ambient laboratory conditions (nominally 70° and 60°F). The lower temperature test was performed by first cooling the sample to approximately 40°F in a refrigerator, placing the sample in the test fixture, and monitoring the sample temperature with a thermocouple. The experiment is initiated when the thermocouple records 60°F, typically about 10 min after removal from the refrigerator. Experiments performed at 40°F were approached in a comparable manner. Thermocouples were not used for the tests at ambient temperature. The test fixture itself was fabricated from aluminum sheet stock, and a schematic is shown in Figure 5.

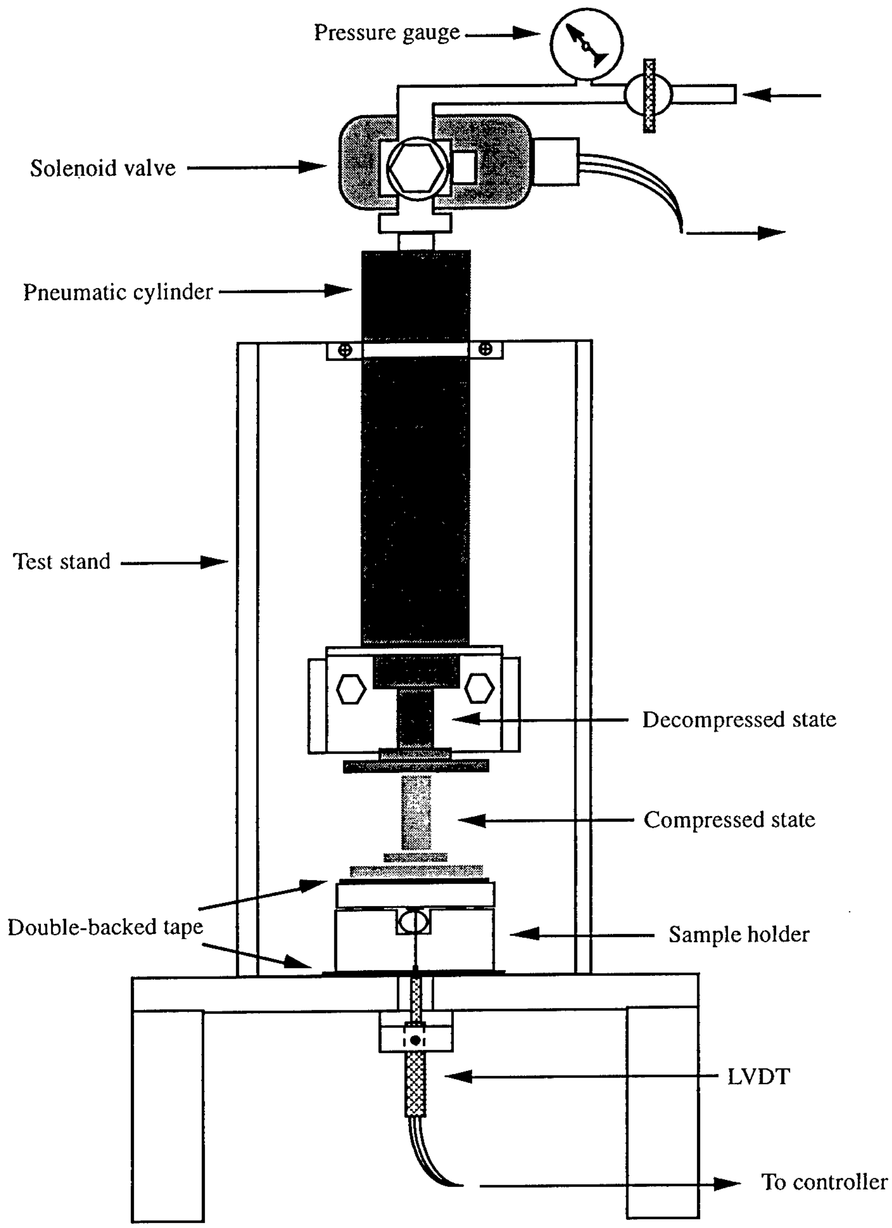


Figure 5. Test stand configuration with sample connected to LVDT assembly (front view).

The O-ring sample was placed in the sample holder assembly so that the protruding end of the metallic strip overhung the holder, and the O-ring itself was flush with the edge of the holder as depicted in the side view of the compressed shimmed sample in Figure 4. An LVDT core was bonded to the metallic strip at the guide hole with epoxy adhesive as shown in Figure 5 (front view) and Figure 6 (side view).

Double-backed tape was used both to constrain the sample holder assembly to the test stand platform and to enable proper centering of the LVDT core into the LVDT coil as indicated by a zero-voltage reading on a digital multimeter. Double-backed tape was also placed on the contact plate of the cylinder rod. The pneumatic cylinder was then pressurized to 30 psi, and the screws holding the upper plate of the sample holder were removed. This procedure retained the original compression on the sample while firmly attaching the sample cover plate to the contact plate of the cylinder rod. The LVDT, which produced an electrical output proportional to the displacement of its separate movable core, was then re-zeroed by means of the adjusting screw indicated in Figure 6. When decompression was initiated, the cover plate of the sample holder was removed from interference with the LVDT

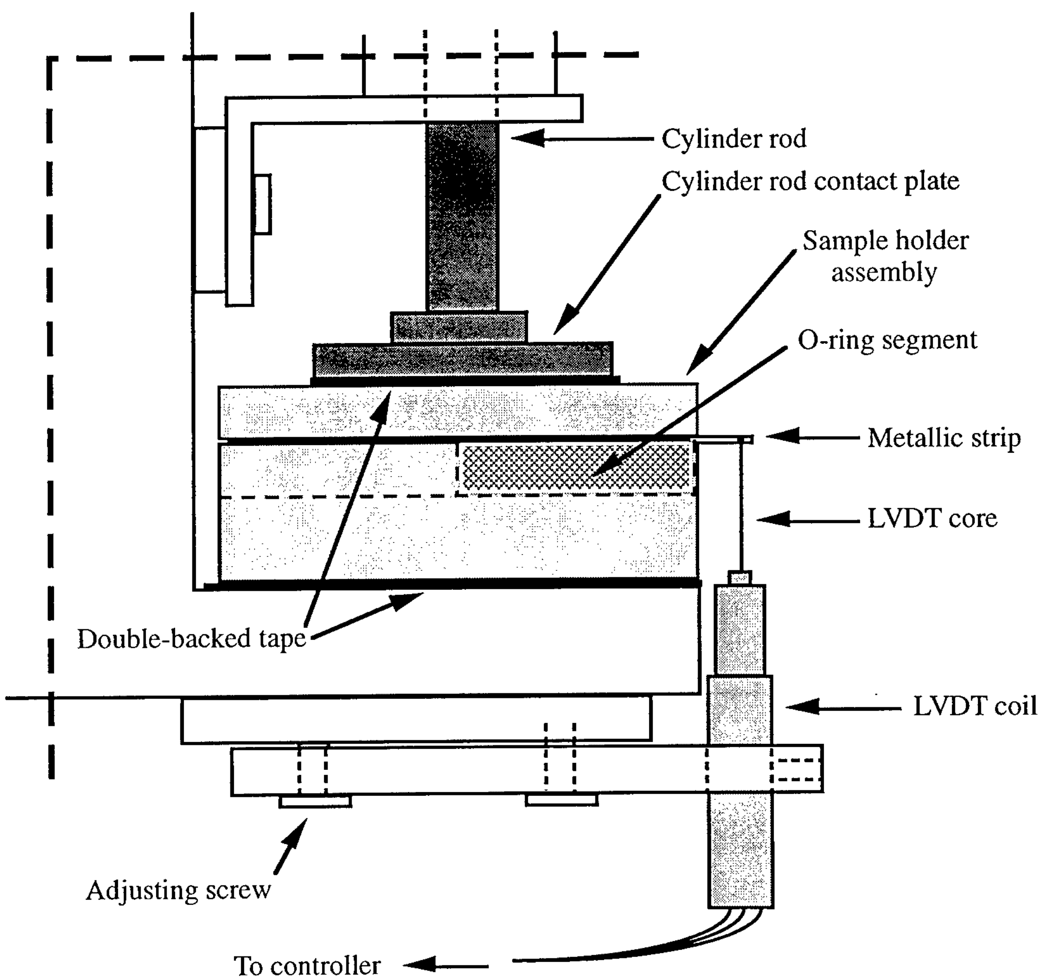


Figure 6. Cutaway section of test stand assembly (side view) illustrating sample-to-LVDT connection.

measurement while the bottom plate was secured from undesirable movement. Before collecting data, it was established that removal of the sample cover plate was faster than the rebound of the O-ring. Thus, the sample cover plate did not affect resilience measurements.

The LVDT assembly was initially calibrated to produce a curve of voltage versus displacement measured in inches. After the components were assembled as described above, a test was performed by initiating a computer program that first included a pretrigger delay of 100 ms, which activated both a computer and recorder. At time zero (0.100 s elapsed time), the solenoid valve was triggered, causing decompression of the pneumatic cylinder, thereby removing the constraining force on the O-ring assembly. (Deviation from smoothness in the data in the first 200 ms is probably the result of uneven expansion of the samples following decompression. Electronic response times of the equipment used to trigger the decompression are on the order of 10 ms and are responsible for any lag in the recovery data observed in the first 20 ms.) Since the displacement of the LVDT core was coincident with the decompression of the O-ring, the dimensional change in the O-ring was transmitted as a change in voltage. The voltage data were collected at the rate of 500 data points per second during a 2-s test period and stored. The voltage data were converted to units of a linear dimension (inches) and changes were recorded versus elapsed time. A test matrix using Viton GLT fluorocarbon elastomer O-ring samples is shown in Table 5.

The Viton GLT samples used in the test matrix shown in Table 5 consisted of samples taken from a single O-ring. The samples were placed under 17% and 23% compression for periods up to 18 months. In addition to the two temperatures used in the previous report (60 and 70°F), some samples were also tested at 40°F.

This report discusses the results obtained for the Viton GLT samples and, where appropriate, will compare these results with those obtained for the Viton B material described in the previous report.[8] Unlike the conditions present in the Titan SRM system, this study was also performed in the absence of any silicone grease used to lubricate the O-ring and the O-ring gland.

Table 5. Compression/Temperature Test Matrix for Viton GLT O-Ring Samples

	17%			23%		
Period	**70°F**	**60°F**	**40°F**	**70°F**	**60°F**	**40°F**
24 hr	x	x		x	x	
1 wk	x	x		x	x	
2 wk	x	x		x	x	
1 mo	x	x		x	x	
2 mo	x	x		x	x	
3 mo	x	x		x	x	
6 mo	x	x		x	x	
12 mo	x	x		x	x	x
18 mo	x	x	x	x	x	x

Viton GLT fluorocarbon samples were cut from a fabricated O-ring that met the specifications for a Titan clevis joint seal, with each sample set consisting of five 1-in. pieces cut consecutively. The average of the five samples used for a specific set of conditions was used to represent the recovery capability of the material under those conditions. A number of contributory factors related to the O-ring samples may not fully represent the absolute response of a complete O-ring configuration. Among these are:

- exposed ends of the samples can deform without the mechanical resistance present in a complete O-ring
- variations in bulk properties throughout the length of a processed O-ring can lead to variations in any average of five samples
- variations in physical attachment of the metallic strip to the O-ring can produce surface area effects
- fixed shim size does not fully meet the dimensions required for the 17% or 23% compression

However, the trends that were produced for the material under study are nonetheless very important in understanding its behavior.

4. Resilience of Viton GLT under 17% and 23% Compression

The studies were performed for different time periods, first for a 24-hr period, and then periodically up to and including an 18-month period. The data were taken for a duration of 2 s, the time allowed for the O-ring to provide sealing capability. Except where indicated, five samples were averaged for each condition. Any deviations of the data from the zero point were found to be due to random electronic noise and not to material response. Errors within each sample set range from a low of 3% to a high of 16%. Deviations from the expected trends in the figures can be attributed to those measurement errors.

Viton B was known to have excellent *compression set properties, described as the percent of deflection by which an elastomer fails to recover after a fixed time under specified squeeze and temperature*. However, its resilience, which is the ability to return quickly to its original shape after a temporary deflection, is compromised by the effect of temperature on its hardness. Results for Viton GLT showed a dramatic difference in resilience from that observed for Viton B under all conditions. Unlike results for Viton B, which showed significantly reduced recovery at 60°F when compared to recovery at 70°F, results for Viton GLT under both 17% and 23% compression appear to show little difference at both 70 and 60°F. The results are shown in Figures 7 and 8 at 17% compression, and Figures 9 and 10 at 23% compression.

Similar to the results observed for Viton B, the rate of recovery for Viton GLT in Figures 7–10 was rapid for the first 100–200 ms before tapering off for the remainder of the test period. Recovery values from 50–70% were observed for the O-ring material made from Viton GLT, significantly higher than those observed for Viton B samples. Values for even the longest time periods under compression in most instances exceeded the highest values observed at the shortest time periods for Viton B samples. These results can be illustrated more dramatically by comparing recovery at the 2-s time frame under comparable conditions for Viton GLT and Viton B samples. Comparative results will be discussed in the following section.

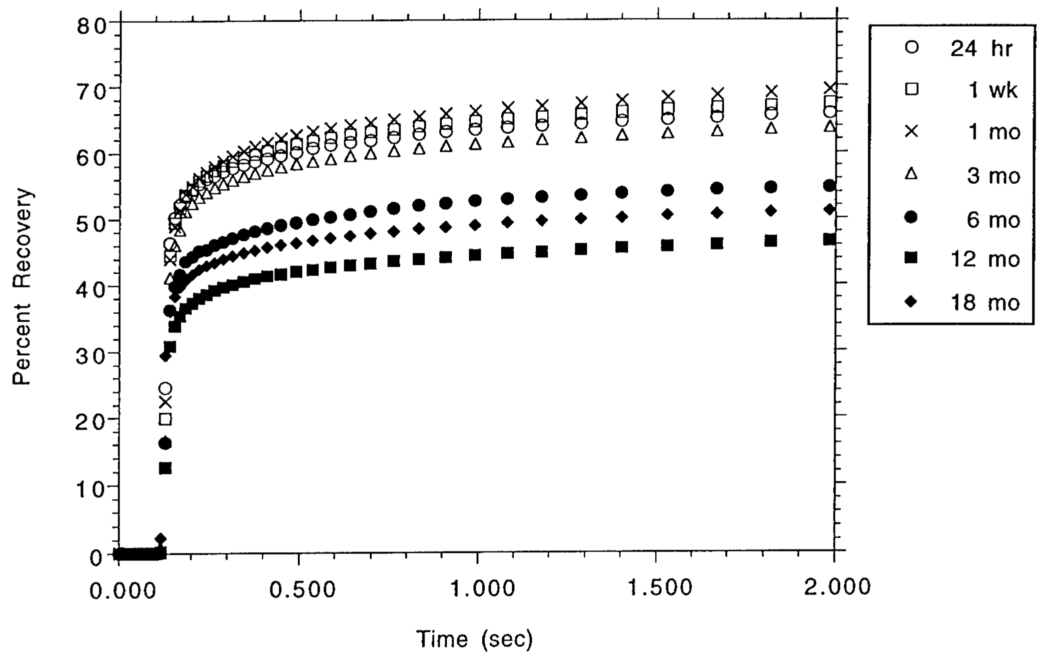


Figure 7. Percent recovery versus time for Viton GLT under 17% compression measured at 70°F.

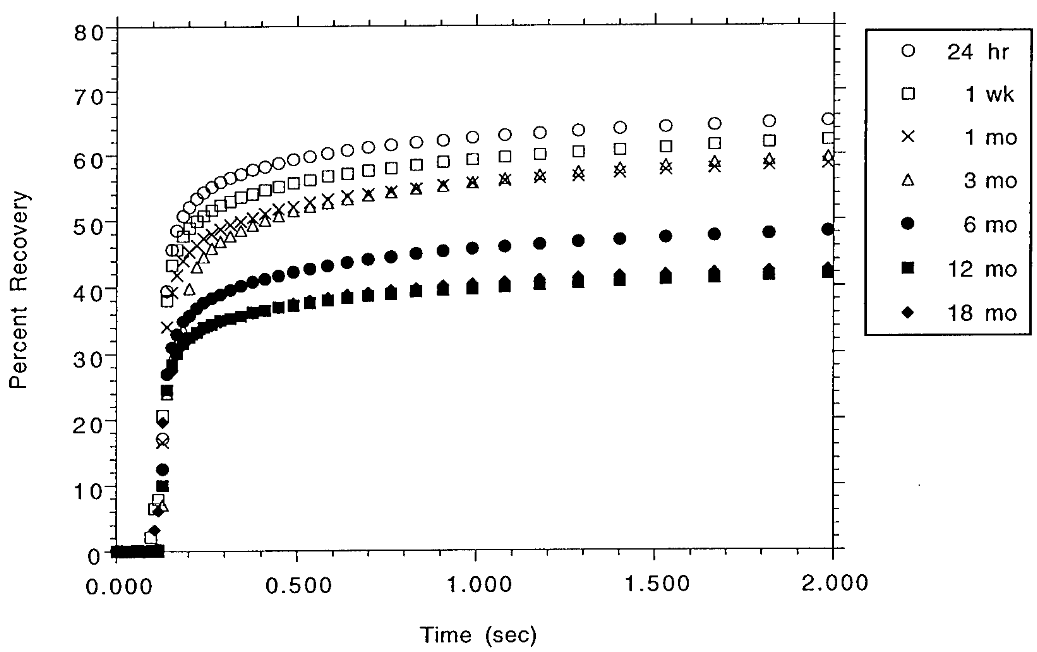


Figure 8. Percent recovery versus time for Viton GLT under 17% compression measured at 60°F.

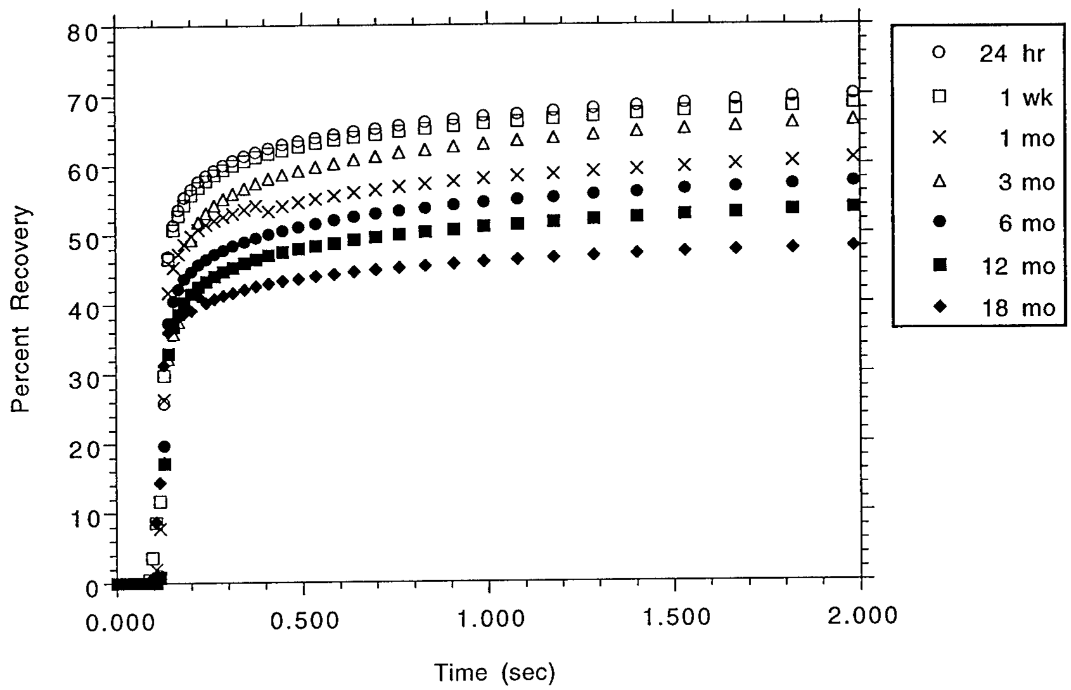


Figure 9. Percent recovery versus time for Viton GLT under 23% compression measured at 70°F.

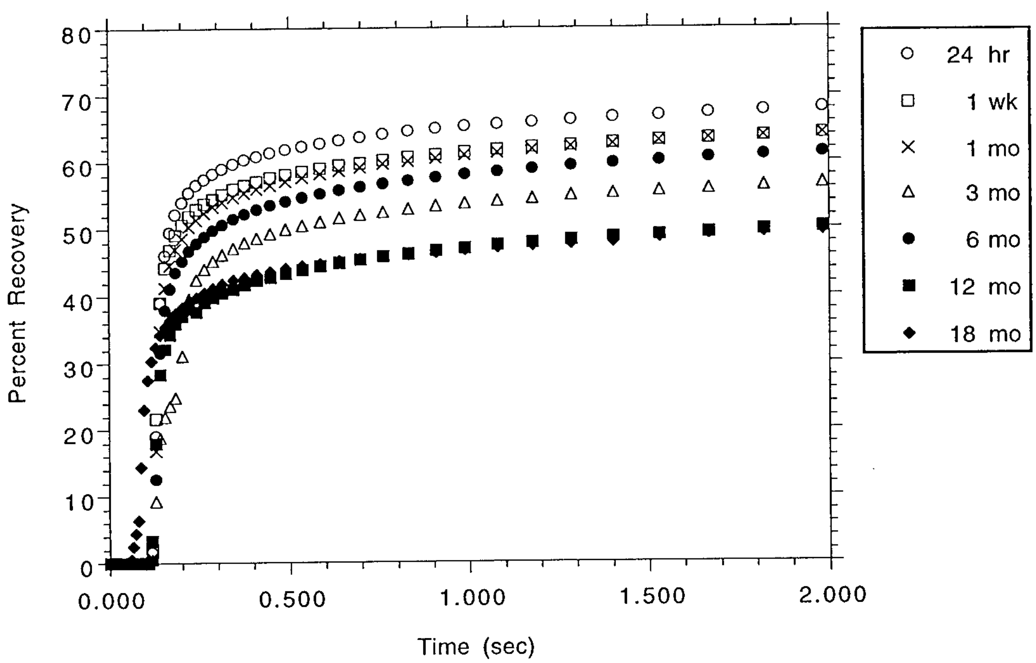


Figure 10. Percent recovery versus time for Viton GLT under 23% compression measured at 60°F.

5. Comparison of Viton GLT and Viton B Resilience Data

Data for Viton B samples at similar times and temperatures were extracted from the previous report[8] and compared with data for the Viton GLT samples measured in this report. Results are seen in Figure 11 at 17% compression and Figure 12 at 23% compression.

Compared over all time periods, the recovery properties of Viton GLT material show significant improvement over that of the Viton B materials studied previously. Although differences can be observed under the two temperature conditions, any reduction in recovery as the temperature is lowered is much less for Viton GLT than for Viton B material. Ordinarily, elastomeric materials become harder as the temperature is lowered. When compared to the other classes of elastomeric materials, Viton B used in the previous study exhibits a significant change in hardness value in the range of 60–70°F.[15] This change in hardness translates to much greater loss in resilience than would otherwise be expected for the very narrow temperature range chosen. This effect is further supported by data describing the dramatic decrease in resilience as the T_g is approached.[16] For Viton B material, the T_g is on the order of –20°C, while that of Viton GLT material is near –40°C, providing much greater temperature latitude for maintaining good resilience properties.

Although the data in Figures 11 and 12 showed some overlapping values, the general trend is for recovery to be reduced with increasing time under compression. Except for anomalous behavior in some data points under compression, the overall results are consistent with the expected order within the variability of the samples that make up the average value. Results observed beyond three months show a much lower rate of change, indicating that most of the compression set occurs early on. Recovery values change little throughout the remaining periods under compression.

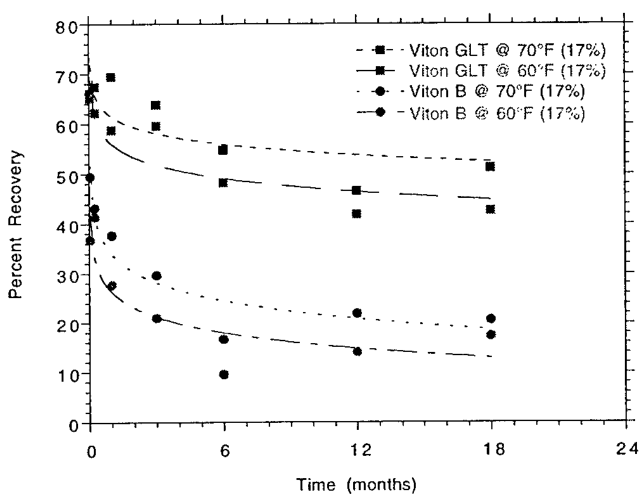


Figure 11. Comparison of total recovery at 2 s versus time under 17% compression for Viton GLT and Viton B measured at 70 and 60°F.

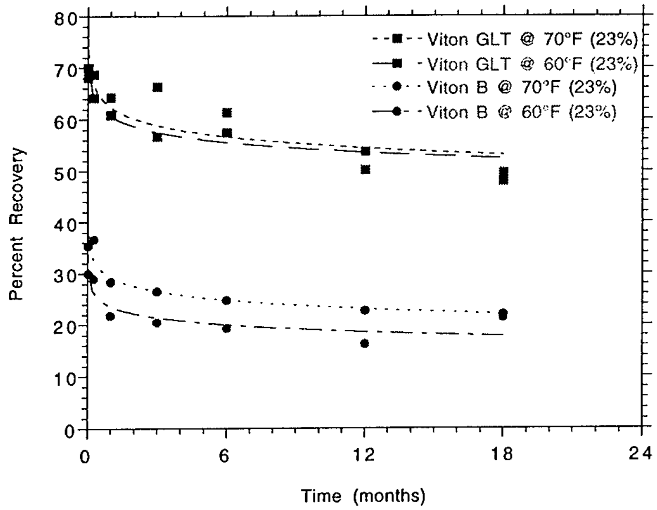


Figure 12. Comparison of total recovery at 2 s versus time under 23% compression for Viton GLT and Viton B measured at 70 and 60°F.

6. Comparison of Viton GLT and Viton B O-Ring Compression Set Properties

Data are presented that relate to ASTM D 395-84 method B, which describes the rubber property of compression set under constant deflection in air.[17] As mentioned previously, this method compresses a test specimen to a specific deflection for a specified time and temperature. The test is usually performed on a cylindrical disc specimen, and its residual deformation is measured 30 min after removal from a compressed state. The compression set is expressed as a percentage of the original deflection, where 0% set constitutes full recovery. Suggested test conditions are 22 and 70 h at 25% compression, under which most reported compression set data are obtained. Although not identical to the conditions described above, compression set data were measured for three O-ring specimens of Viton B from a previous study and the Viton GLT specimen in this study. Results for samples measured after one week under compression at 70°F are shown in Figure 13.

Results in Figure 13 reveal the significant difference in elastomeric properties that can be conferred on a fluorocarbon material through variations in formulation described earlier. The Viton GLT formulation has significantly better compression set properties under the measured conditions than does the Viton B material. Results in Figure 13 are also consistent with reported compression set data for Viton B. Considerable batch-to-batch variability was measured among the three O-ring samples for Viton B. This variability translates to variation in resilience properties. In general, the recommended

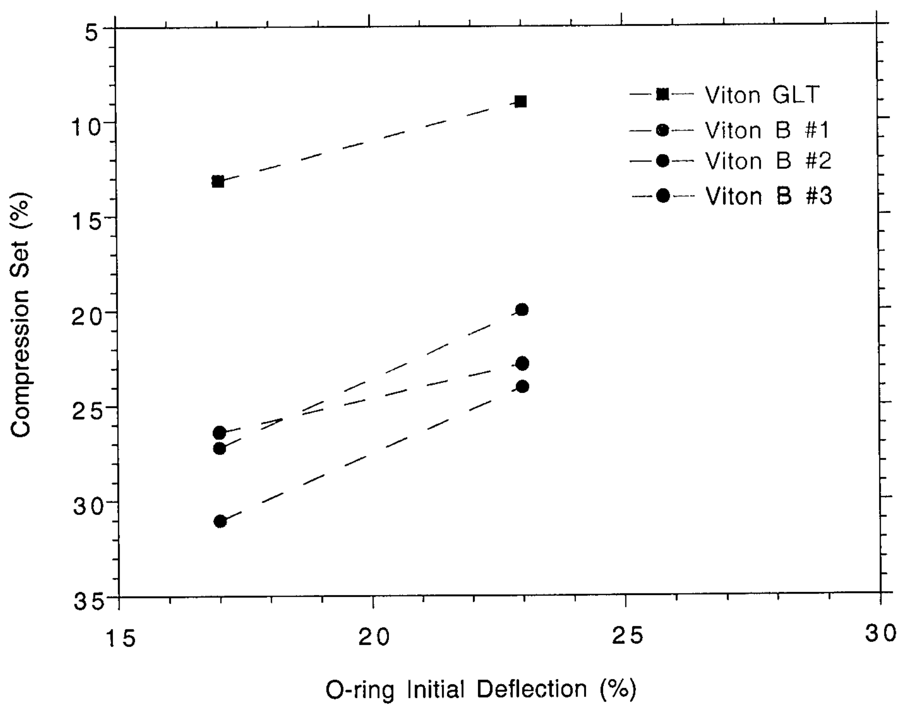


Figure 13. Comparison of compression set data for one Viton GLT batch and three Viton B O-ring batches after one week at 70°F.

compression (squeeze) for seals is on the order of 15–30%. It has been reported that for deflections <15%, the compression set results are unreliable. Conversely, at deflections of >30%, the extra stress can result in premature seal failure.

These measurements were obtained in the absence of any lubricant. A comparison of lubricated and unlubricated Viton B O-ring seals is contained in the previous report.[8] Results indicated, at least through a 12-month period, that lubrication was not a significant factor in O-ring resilience. Other studies involving O-ring lubrication under a variety of conditions have produced mixed results.[5,18,19] Lubricants can affect the properties of O-ring seals either by shrinkage or swelling. Either result can affect the compression set and resilience properties of the O-ring seals.

7. Viton GLT O-Ring Resilience at 70, 60, and 40°F

With the remaining Viton GLT material, additional data points were obtained at 40°F for the 12-month time period under 23% compression, and for the 18-month time period under both 17% and 23% compression. Recovery data were first assembled for the 12-month period at 70, 60, and 40°F, and the results compared in Figure 14.

After 12 months under compression, recovery at 70°F is shown to be nearly 54% after 2 s, only slightly reduced to 50% at 60°F. Measurement at the colder temperature (40°F) lowered the recovery value to 40%. Given a reduction of some 25% from the value at 70°F, the resilience for Viton GLT is remarkable when compared with the earlier data for Viton B.

The results for the three temperature conditions taken for the 18-month period under two compression conditions also follow the expected trends described previously. The results are shown in Figure 15 for 23% compression and in Figure 16 for 17% compression.

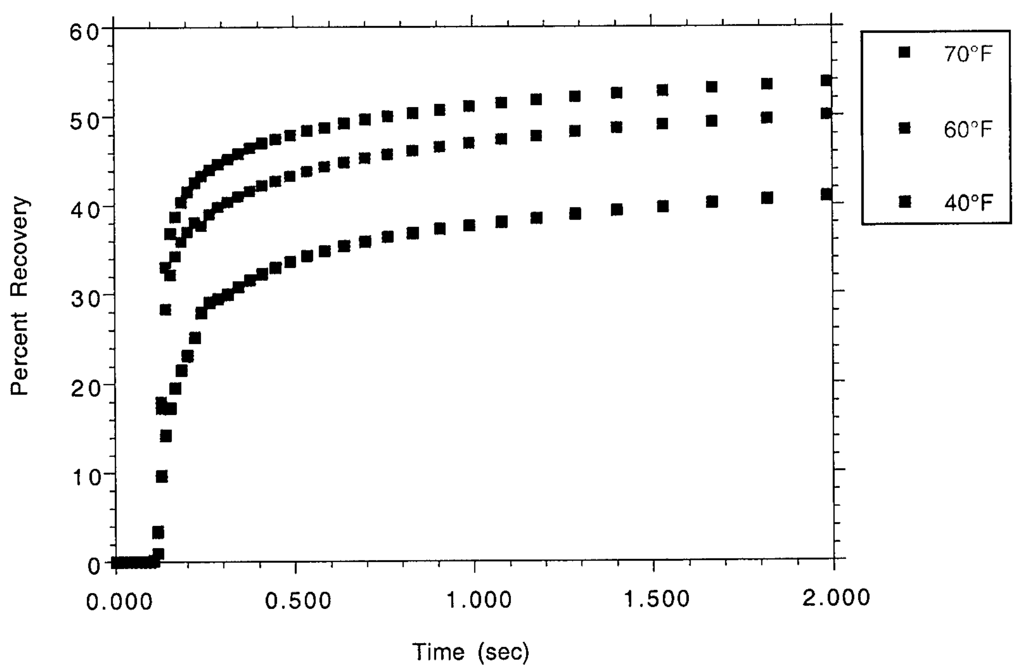


Figure 14. Comparison of percent recovery versus time for Viton GLT samples under 23% compression for 12 months, measured at 70, 60, and 40°F.

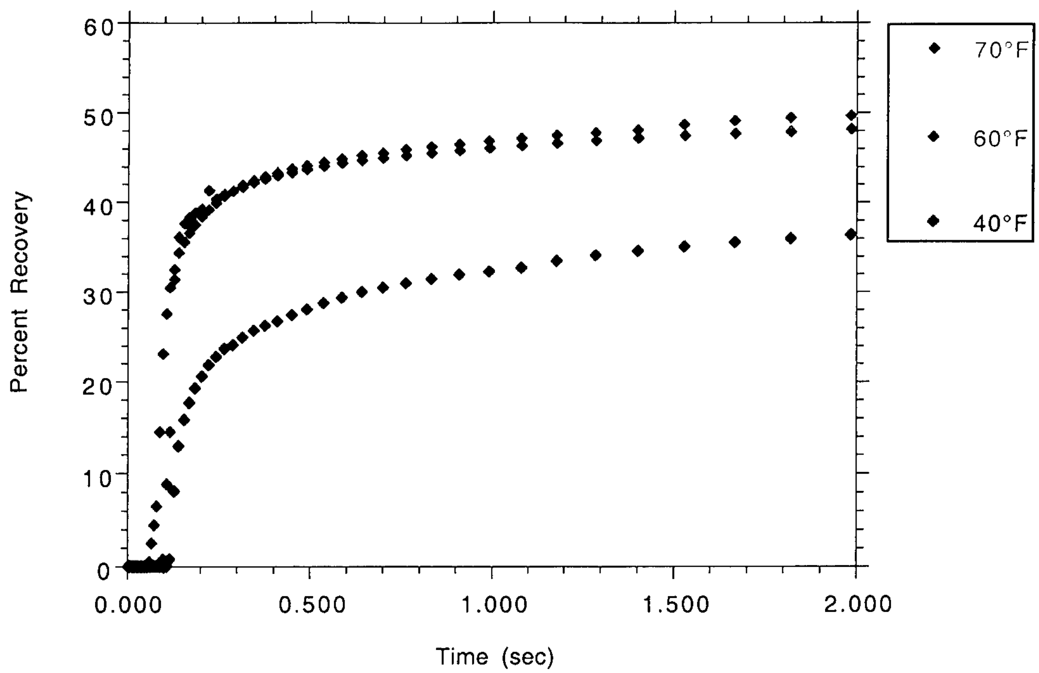


Figure 15. Comparison of percent recovery versus time for Viton GLT samples under 23% compression for 18 months, measured at 70, 60, and 40°F.

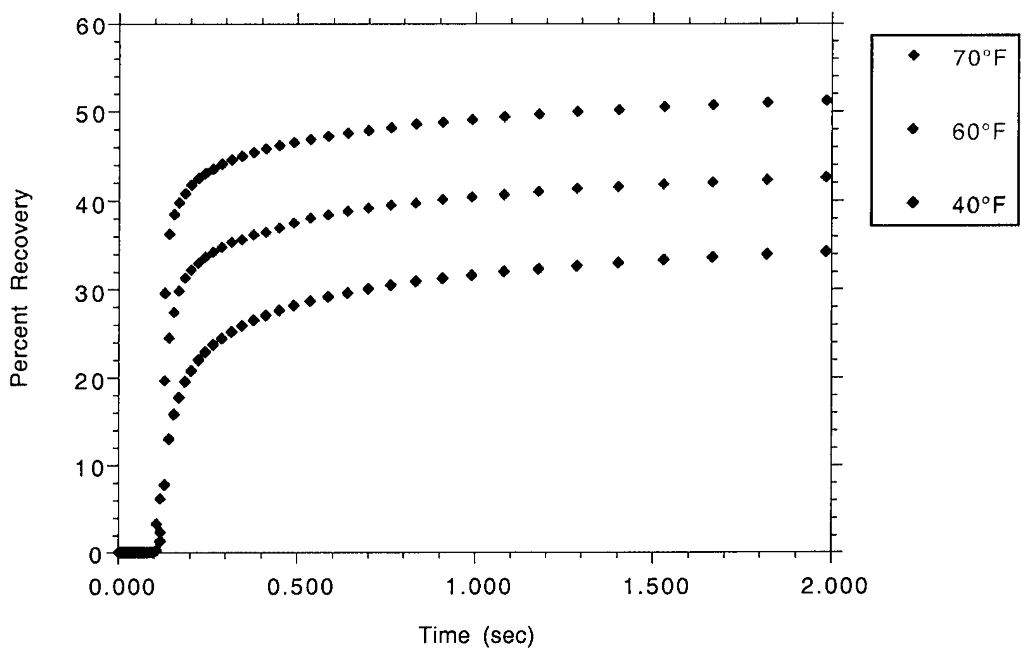


Figure 16. Comparison of percent recovery versus time for Viton GLT samples under 17% compression for 18 months, measured at 70, 60, and 40°F.

After 18 months under compression the results for Viton GLT in Figures 15 and 16 indicate significant low-temperature capability when compared to Viton B. Even at 40°F, Viton GLT surpasses the results observed for Viton B at the higher temperatures measured for resilience. By inherently exhibiting greater resilience, the Viton GLT material would provide greater capability at lower temperatures than would Viton B.

8. Discussion

Resilience measurements of Viton GLT O-ring material under compression for up to 18 months showed an initial rapid rate of recovery in the first 100 ms, diminishing significantly during the next 300 ms, and essentially leveling off through the end of the 2-s test period. The effect of temperature on resilience for Viton GLT was much less significant than that observed for Viton B, and these properties were maintained to temperatures as low as 40°F.

There are a number of differences between the O-ring material test fixture used in this report and that of the launch vehicle environment that could affect resilience:

- use of short O-ring sections
- constraint at the top of the O-ring by the bonded tab
- variability in the viscoelastic properties between processed O-rings
- O-ring lubrication

A full-scale simulation would use an entire O-ring, eliminating the unconstrained open-face edge effects present in the linear sample configuration. With an aspect ratio of only 4:1 in the linear sample configuration, the effect of compression on resilience may deviate from that of the O-ring configuration. Because the ends of the samples are unconstrained, O-ring resilience values in the direction of decompression may not be fully realized. Although the linear sample configuration may result in lower values, the differences are not expected to be significant at this aspect ratio.

The O-ring samples are also bonded to a metal tab for attachment to the LVDT core. This attachment is confined to a small surface area of the O-ring, and its impact on resilience is also not considered significant.

The variability in the samples that make up the average for any time period can also be the result of inhomogeneities in the O-ring, which is not unusual for a filled system and the small sample size used. Because the viscoelastic properties of a whole O-ring should average out such inhomogeneities under decompression, the average of the individual sample values should be more representative than any one specific value.

The application environment about the O-ring can also affect its resilience. In the former SRM application procedure, the O-ring was coated with silicone grease and installed in the steel gland. The remainder of the gland space was filled with the same grease. Advantages of lubrication include facile O-ring mobility under dynamic compression, reduced chemical interaction between O-ring and gland materials, and partial swelling of the O-ring material that serves to enhance resilience. The Vitons are highly resistant to fluid degradation, primarily exhibiting variation in swelling

characteristics. Any new O-ring material should be subjected to compatibility studies that eliminate any unexpected swelling characteristics as a factor.

An additional concern is possible corrosion at the contact point between the O-ring and the steel, and the effect of this interfacial layer on the mechanical properties of the O-ring. As stated earlier, adhesion characteristics between Viton O-rings and metal surfaces depend primarily on the curing system used. Du Pont literature states that diamine-cured fluorocarbon elastomers provide better adhesion than those that are bisphenol- or peroxide-cured. Only qualitative information is available to support this observation. Separate studies on the effect of lubrication on O-ring compression recovery gave mixed results. However, it appears that the lubricated surfaces may tend to minimize surface interactions that can occur, thereby reducing potential for loss of O-ring resilience from an external effect.

The differences in the O-ring test environment described in this report compared to that of the launch vehicle environment are expected to have minimal impact on resilience. The data presented here, supported by comparable compression set and resilience characteristics for O-rings published elsewhere, are expected to closely represent resilience values that would be exhibited during launch.

9. Conclusion

O-ring material made with Viton GLT was found to have significantly improved resilience and compression set properties under all conditions studied when compared to Viton B. Even under worst-case conditions, these properties exceeded even the best values obtained in a comparison study with Viton B. Based on these observations, Viton GLT can be considered to be an excellent candidate for use in demanding applications that require these desirable elastomeric properties. Both Viton B and GLT families of fluorocarbon elastomers, although of different backbone structure, are high-performance materials capable of excellent thermal stability and fluid resistance, and the ability to seal against hard vacuum. Resistance to compression set in Viton GLT is among the highest of all rubber materials, with the difficult-to-measure resilience properties also considered to be excellent. However, compatibility concerns that arise where O-ring materials come in contact with external environments should always be addressed before recommending its use in new or existing applications.

References

1. Rogers, W. P. (Chairman), *The Report of the Presidential Commission on the Space Shuttle Challenger Accident*, U.S. Government Printing Office, 1986.

2. Lach, C. L., "Effect of Temperature and O-Ring Gland Finish on Sealing Ability of Viton V747-75," NASA TP-3391, NASA Langley Research Center, 1993.

3. Flitney, R. K., B. S. Nau, and D. Reddy, *The Seal Users Handbook*, 3rd ed., **36**, 1994.

4. Stein, S. "Seal Material Selection, Design and Performance—Advancements from the Space Shuttle Booster Redesign," Morton Thiokol, Inc., AIAA-89-2774, AIAA/ASME/SAE/ASEE 25th Joint Propulsion Conference, 10–12 July 1989.

5. Wensel, R., and L. Welch, "Factors Affecting Compression-Recovery of ASRM O-Rings," AECL Report, ET-S-357, ASRM Report, SE16133, Chalk River Laboratories, October 1992.

6. *Military Standardization Handbook Rubber Products: Recommended Shelf-Life*, MIL-HDBK-695C, AMS 7259, 27 March 1985.

7. Wood, T. E., and W. P. Stone, "Effect of Shelf Aging on O-Ring Materials," NASA TM-78150, George C. Marshall Space Flight Center, January 1978.

8. Giants, T. W., "Viton® B O-Ring Resilience Study," TR-99(1413)-6, The Aerospace Corporation, January 2001.

9. "Viton® Selection Guide," H-41668-1, The Du Pont Company, 1993.

10. *Encyclopedia of Chemical Technology*, 3rd ed., **8**, 500–515, 1979.

11. Miller, G. A., and L. H. Sperling, *Polymer Engineering and Science*, **22**, (8), 478–483, 1982.

12. Arnold, R. G., A. L. Barney, and D. C. Thompson, *Rubber Chem. Technol.*, **46**, 619–652, 1973.

13. Schmiegel, W. W., *Die Angewandte Makromolekulare Chemie*, **76/77**, 39–65, 1979.

14. *Encyclopedia of Polymer Science and Engineering*, 2nd ed., **7**, 256–269, 1987.

15. *Parker O-Ring Handbook*, ORD 5700, Parker Hannifin Corporation, A3–A10, 1992.

16. Stacer, R. G., D. M. Husband, and H. L. Stacer, *Rubber Chem. Tech.*, **60** (2), 227–244, 1987.

17. Standard Test Methods for Rubber Property—Compression Set, *Annual Book of ASTM Standards*, ASTM D 395-89 (Reapproved 1994), **09.01**, (9), 1977.

18. McMurtry, W. M. and G. F. Hohnstreiter, "Determination of Spring Modulus for Several Types of Elastomeric Materials (O-Rings) and Establishment of an Open Data Base for Seals," SAND-95-0373C, Sandia National Laboratories, 1 January 1995.

19. Wensel, R., and K. Wittich, "Development of an Improved Elastomer for the ASRM O-Rings," AECL Report, ET-S-340; ASRM Report, SE16075; Chalk River Laboratories, June 1992.

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